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**Subject:** Primary Processes R & D
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**From/Location:** E. L. Cambridge/J. C. Withers

**To/Location:** J. G. Kaufman

## BASIC RESOURCES RESEARCH

### Bauxite Evaluation Capability

Jamaican and Brazilian bauxite as well as NBS standard samples have been received. A method for analyzing for $P_2O_5$ has been tested and found to be acceptable. A digestion bomb has been set up to digest and extract alumina from the bauxite samples.

### AD-120-$AlCl_3$ Process

Further confirmation has been obtained that our air treatment process for green coke results in $AlCl_3$ with less than 3 ppm of PCB's. Confirmation has also been obtained that green coke and ACH treatments reduce the hydrogen content to acceptable levels of less than 0.5 percent and 0.2 percent respectively. Although pretreatment for ACH has not been optimized for reaction kinetics, a mass transfer number of

$$16 \frac{\text{kg mole } AlCl_3}{\text{hr } M^3 \text{ Atm}}$$

has been achieved in the scaled up two-inch diameter fluid bed reactor. This mass transfer number contrasts to the Alcoa mass transfer number, which is reported as

$$7 \frac{\text{kg mole } Cl_2}{\text{hr } M^3 \text{ Atm.}}$$

Since we are reporting with reference to $AlCl_3$, the number can be multiplied by 1.5 for comparison to the $Cl_2$ Alcoa number. Thus, our 16 number is comparable to 24 on the Alcoa scale.

Double calcining treatments for ACH that include initial rotary kiln at 300°C followed by vacuum and gases $H_2$, $CO_2$, $Cl_2$ and $NH_3$ at temperatures up to 1000°C, did not reduce the hydrogen level over that achieved by the standard treatment in air up to 700°C. The residual $Cl_2$ and $H_2$ were higher with the $NH_3$ cover gas. Trihydrate was also treated in these atmospheres for comparison and to prepare different aluminas for solubility studies in modified Hall electrolytes.

A statistical design set of experiments between Anaconda produced ACH and USBM ACH showed that at lower temperatures the Anaconda ACH had about 1 percent less residual $Cl_2$ than USBM with no difference in $H_2$ at lower or higher temperatures. The statistically most important parameter for residual $Cl_2$ or $H_2$ is temperature.

The NASA computer code has been expanded to include $AlCl_3 \cdot 6H_2O$ and its possible decomposition compounds. When applied to the calcination of $AlCl_3 \cdot 6H_2O$ the results show that it is thermodynamically possible to produce $Al_2O_3 \cdot H_2O$ (boehmite) in the temperature range of 127 to 230°C. The literature shows that boehmite can be calcined to the highly reactive $\gamma$-$Al_2O_3$ at 450°C. Accordingly, we are investigating the possibility of converting $AlCl_3 \cdot 6H_2O$ in a two-step calcination to very high surface area $\gamma Al_2O_3$.

The calcining treatment of green coke in air results in reducing PCB levels in $AlCl_3$ produced with the treated coke as the reductant to less than 3 ppm (2788 ppm not treated to 2.6 ppm treated) has been reconfirmed. However, using fully calcined coke which was treated in air similar to the green coke, did not significantly reduce the PCB level (45 ppm to 35 ppm). A record of invention is being prepared for the green coke treatment process.

It is necessary to calcine the ACH to 700°C to obtain acceptable $H_2$ levels. Comparative chlorination rates between calcining at 400°C and 700°C show no reduction in rate which is in contrast to the CSIRO patent and considered an unexpected improvement.

The economics and technical performance of the reductant CO and green coke are being continually compared for the production of $AlCl_3$. If CO is the ultimate reductant, we must be assured of an economical uninterrupted supply. Consequently, we have undertaken a literature search and cost comparison of possible processes to produce CO. Forty-six coal gasification processes have been identified which meet all our criteria. Costs have been received for only two thus far, which are 6 and 10 cents per pound CO. Our previous economic model showed 7 cents per pound as a break-even with coke. The economics model for all sensitivities of the $AlCl_3$ process is being updated.

The Anaconda fluoride based refractories are being tested in the Alcoa bipolar salt electrolyte. In 30 hours of cumulative tests, there is no difference between our refractories and Sialon.

An interim progress report is being prepared. The expected issuance date is the first week of June.

## REDUCTION RESEARCH

### Ad-108 Composite Anode Process

The powder anode concept was further investigated this month. The powder was made of crushed prebaked composite anode with particle size distribution of -20 to +12 mesh. A boron nitride (BN) cup with drilled holes to give up to 42 percent pore structure was used as the nonconductive porous diaphragm to hold the powder. A graphite rod was utilized as a current collector. Current-voltage sweeps were recorded in all fluoride melts. The results indicate that only very low current density can be achieved without excessive voltage losses. This infers high contact resistances between the current collector and powder particles and also between individual particles. The results also establish that the electrochemical reaction at the anode is

definitely the $CO_2$ evolution, with no anode effect. This infers that the concept of the composite anode works, however, the powder approach results in excessive cell voltage due to the high resistivity of the powdered anode.

In another set of experiments utilizing the powdered anode, current-voltage curves were obtained in mixed chloride-fluoride melt (the chloride salt was added to increase wettability of the composite powder). The results obtained so far, support the above finding that only unacceptably low current density can be achieved without excessive voltage loss.

A new diaphragm design with over 90 percent open pore structure and current collector technique is being investigated. If this design does not result in acceptable low voltages at high current density, work on the powder anode approach will take lower priority than prebake and other anode configurations.

A new die for hot pressing pre-baked composite anodes was designed and constructed. It has undergone several modifications in trial operations. The die now appears to work sufficiently well to begin more detailed anode development. A statistically designed set of experiments for composite anode formulation will be conducted using this new die.

Test runs have been made utilizing the self-baked composite anode concept. Difficulty was encountered with pitch oxidation. New test runs are being conducted with the self-baked composite anode in a configuration that prevents pitch oxidation.

Experiments are underway to determine a materials balance for a small (100 amp) composite anode cell. Initially, the compositional change of molten salt electrolyte without the composite anode electrolysis is being monitored to obtain the background data needed to correct for melt composition during anode electrolysis. More than one week duration has been logged. Analytical results of salt composition should be available shortly.

AD-116 Potlining Resource Recovery

A report presenting the objective, methods, results and conclusions of the joint Alcan/Anaconda pre-phase one study is being prepared by Alcan engineers. It was concluded, at the April meeting in Montreal, that a Phase One study should be undertaken to investigate the Alcan Mini-L and Anaconda-116 processes so that process parameters could be sufficiently defined to:

(1) Select or design a mutually exclusive flowsheet for resource recovery;

(2) Design a Phase Two pilot plant facility.

The estimated time and expenses for the Phase One study is six man-years and $150,000 out-of-pocket expenses. The Alcan pre-phase one report is expected to be completed in early June.

Petroleum Pitch

Achieving a good baked density for composites utilizing Ashland-240 pitch remains a problem at Columbia Falls. The effort has been suspended due to other priorities and will be undertaken at the new Carbon Lab in Tucson.

Tucson Carbon Work

Examination of Columbia Falls coke and bath skim has tentatively revealed that binder factors are more responsible for the dusting problems than the filler coke quality.

Experimental work is scheduled to begin on Columbia Falls anode optimization about June 1 in the new lab.

Following a request from Dr. Bartlett, our conclusion is that Australian brown coal is not suitable for anode carbon. Domtar, Inc. of Ontario, Canada can supply up to 50,000 pounds per year of coal-tar pitch with any adjusted QI in the form of liquid or flake.

Consulting consultation is being given Columbia Falls on each of their carbon programs. Sam Jones spent the week of May 3-7 at Columbia Falls reviewing and consulting in overall carbon operations.

DEVELOPMENT & TECHNICAL SERVICES

Lithium Fluoride

The Sebree testing was terminated in April with the shutdown of Line 2. No further experimentation is planned at this time.

In the Columbia Falls test, lithium fluoride concentration is at 0.9 percent in the five test cells. Temperature of the cells has increased 8-10°C, but no operating problems have been noticed. Increased additions of lithium carbonate will continue until the target of 2 percent lithium fluoride is reached.

The techno-economic assessment reports for Columbia Falls and Sebree are completed. A trip to Columbia Falls is scheduled in early June to discuss with plant personnel results of the study and possible additional testing.

Columbia Falls

Cooling manifolds for the magnetics probe was tested at Columbia Falls in the Bus Modification Program. A temperature of 300°C was achieved in the vicinity of the probe location. More work is being done to reach the required temperature of 75°C.

Bus modification to the first pot is scheduled for July. A total of four pots are planned to be modified in 1982.

Action plans for the three phases of the Anode Optimization Program were developed. Responsibilities and milestone dates have been set to complete each task. Phase I testing is scheduled to begin June 1st.

Sebree

Sam Jones and Subodh Das are currently writing a proposal similar to that for Columbia Falls work for Sebree. The proposal is expected to be completed by the end of May for review by Sebree management.

A feasibility study on the synergy of plant operations with a 24-day anode is in progress. The report is planned to be completed in early June.

FACILITIES

The Carbon and Reduction Research Laboratory is complete less some punch-list items. It was formally accepted from the contractor May 14. After acceptance and testing the floor for pitch absorbency, it was decided to apply additional floor sealing to be completed May 22, 1982. Formal move-in will begin May 24. With over 60 percent of the cost accountable, we remain about 6 percent under budget for equipment purchases.

PERSONNEL

Ed Creamer from Argonne National Laboratory has accepted a position as Staff Research Engineer in Reduction; Bert Bell has accepted a transfer offer from Louisville to Alumina and Chemicals as a Staff Research Engineer, and Kirk Weisbrod has accepted a transfer offer from Anaconda Minerals in Plano as a Scientist in Carbon and Materials. Other interviewing has taken place and it is anticipated that additional hiring will result.

Jim Withers

J. C. WITHERS

JCW:pm

cc: R.W.Bartlett
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